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CHARACTERISTICS OF A "HIGH SOLIDS" NEBULIZER FOR FLAME ATOMIC ABSORPTION SPECTROMETRY

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CHARACTERISTICS OF A "HIGH SOLIDS" NEBULIZER FOR FLAME ATOMIC ABSORPTION SPECTROMETRY

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ABSTRACT

Studies are presented to characterize a new nebulizer developed for direct atomic absorption analysis of extremely complex clinical and environmental materials. Important parameters are described concerning the design and performance of "high solids" spectrochemical nebulizers based on the *Babington principle*.

INDEX HEADINGS: Nebulizer; Direct determination; High solids analysis; Atomic absorption.

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INTRODUCTION

"Clog free" production of high density, finely dispersed aerosols from solutions and slurries of elevated salt concentration, high solids content, and increased viscosity has recently been described for spectrochemical sample introduction by Fry and Denton (1). The Babington principle of aerosol generation (2,3) was adapted for direct atomic absorption analysis (using high solids burner heads) of samples as complex as sea water, urine, whole blood, evaporated milk concentrate, and tomato sauce (1). The superior tolerance of this approach to "high solids" slurries is a direct result of eliminating the conventional requirement that the sample must pass through a small bore capillary tube. Ability of the material to pass through the relatively unrestricted sample delivery pumping system was found to be the primary limitation on the complexity of materials that can be directly analyzed (1) by the new procedure. The improved sample handling capability is achieved without significant sacrifice of precision or concentration sensitivity when compared with conventional capillary pneumatic approaches (1). This relatively "preparation free" sample introduction technique for analytical atomic spectroscopy can presently be considered to be in the beginning stages of development. Once design and operational characteristics are well established, this convenient approach to high solids atomization holds great promise for application in agricultural, clinical, environmental,

and industrial analytical situations. The purpose of this paper is to outline design considerations and characterize the parameters that govern the performance of this new high solids sample introduction approach at its present state of development.

I. EXPERIMENTAL

A. Apparatus. A previously described (1) atomic absorption spectrometer, 10 cm. high solids single slot air-acetylene burner head, 10 cm triple slot Boling burner head, Varian Techtron (Springvale, Vic., Australia) and Jarrell Ash (Waltham, Mass.) capillary pneumatic nebulizers, Beckman (Fullerton, Calif.) total consumption burner operated as premix nebulizer, "HYDROSPHERE" (Babington principle) model respiratory nebulizer system (Owens Illinois - Toledo, Ohio), and specially developed Babington principle nebulizer chamber (cylindrical) with variable flow peristaltic pump sample delivery system were used in the course of these investigations. Quick interchange connections to the premix chamber were provided for the nebulizer mountings. Provision was made in the cylindrical nebulizer chamber for accommodation of a variety of configurations for aerosol generation involving the Babington principle. Fig. 1 diagrams the "HYDROSPHERE" system with a slight modification; other configurations constructed and evaluated in this study are illustrated in Figs. 2 - 5. All configurations shown represent adaptations of the Babington principle which involves flowing a thin

film of sample over the surface of a pressurized sphere or hemisphere insert. Interruption of the sample film at the point of an orifice by the high velocity exit of oxidant gas serves to generate the aerosol. Since the sample is not required to pass through this orifice, clogging does not occur even for the most complex materials (See Fig. 6) when the systems diagrammed in Figs. 2 and 5 are used. Small ($\leq$ 5μm) aerosol droplets (4) are carried by the gas stream to the premix chamber of the burner. Larger droplets and sample runoff from that portion of the sample film not flowing across the orifice either exit via the drain (Figs. 2, 5, and 6) or may be collected for recirculation (Fig. 1, Fig. 3, and Fig 4). Recirculation was evaluated in several configurations for the purpose of conserving solution. The glass nebulizer spheres indicated in Figs. 1 - 4 were adapted from the Hydrosphere system formerly available from Owens Illinois (Toledo, Ohio) and presently available from McGaw Respiratory Therapy (Irvine, Calif.). The compression fitting design of Fig. 5 represents a convenient prototype approach allowing rapid interchange of a variety of improved metal nebulizer inserts. The 6 mm insert was constructed by rounding and polishing one end of a 6 mm o.d. rod using an engine lathe. The rod was then cut to a length of 19 mm and drilled from the"cut" end at a diameter of 5.5 mm to a depth of 18.5 mm using care to avoid drilling through the rounded end. The desired orifice was machined in the side of the rod 3 mm below the

rounded end. The 3 mm inserts were constructed in similar fashion utilizing a 6 mm base and 2.5 mm i.d. dimension with the orifice machined 1.5 mm below the rounded end. The inserts are seated conveniently in the modified compression or Swagelock (Solon, Ohio) fitting holder. Non-wetted materials such as Teflon are not recommended for the nebulizer insert since aqueous samples tend to bead up on such surfaces rather than form a suitable film. Following initial cleaning, the flowing sample and solvent stream keeps the nebulizer insert sufficiently clean to maintain a thin film flow of material indefinitely (the nebulizer insert in this laboratory has not required cleaning in over a year for all high solids samples analyzed (1)). The spray chamber walls should, however, be periodically rinsed (with disinfectant if desired) following analysis of large numbers of biological samples such as blood to prevent buildup and spoilage.

B. Procedure. The atomic absorption of cadmium at 228.8 nm was utilized to study the effect of parameters governing nebulizer performance. The Cd hollow cathode was operated at 5 ma, the spectral bandpass was 0.6 nm, and the instrumental time constant (5 RC) was 0.6 sec. Cadmium was chosen for its simple spectrum, the high volatility of its salts, and its freedom from oxide formation and ionization effects. These properties make the atomic absorption of this element in a 10 cm premixed air-acetylene flame relatively independent of

instrumental parameters such as redox character and viewing position in the flame. Nebulizer performance under a wide range of conditions was thereby more readily and independently assessed. The nominal viewing position was 6 mm (beam center) above the burner head. Whenever oxidant (nebulizer) gas flow rates were varied, fuel adjustment was made to achieve a constant flame stoichiometry. Studies were also performed to ensure that the vertical absorbance profile had not been significantly altered. Oxidant gas flow rate measurements were corrected for pressure drop across the nebulizer orifice. Volumetric determination of aspiration rates and pump delivery rates were made when appropriate.

The sample delivery tube and corresponding inlet to the cylindrical chamber (Figs. 5 and 6) were threaded to facilitate external adjustment of the sample delivery gap (distance between the end of the delivery tube and the top of the pressurized nebulizer hemisphere).

II. RESULTS AND DISCUSSION

A. Operating Range. A series of Babington nebulizer inserts including 0.13 mm, 0.34 mm, 0.61 mm, and 0.71 mm single hole orifices were constructed and the resultant operating range of oxidant flow rate evaluated. The 0.13 mm and 0.34 mm orifices produced extended low flow rate operation (down to $\sim$ 1 L/min). The wide dynamic operating range of these orifices also extended to the normal operating range of slot burners ($\geq$ 5 L/min), but pressures in excess of 100 psi. were required

to drive the nebulizer in this higher range. The 0.71 mm insert orifice was found to operate at much higher flow rates (> 10 L/min) with reduced pressure requirements, however the sensitivity was degraded and the operating range did not extend sufficiently low enough. The 0.61 mm orifice optimally covered the normal (8-15 L/min) slot burner range (Fig. 7) using pressures of 14-40 psi, so the other orifices (0.13 mm, 0.34 mm, and 0.71 mm) were not considered further in these studies.

Although Fig. 7 bears out the previously reported (1) sensitivity ntage of the Varian Techtron nebulizer (when analyzing "clean" aqueous solutions), the metal version of high solids nebulizer diagrammed in Fig. 5 is seen to have a superior operating range of oxidant flow rate (Fig. 7). The effects of Fig. 7 are primarily related to nebulizer performance. Aspiration rate measurements indicate that the Varian Techtron nebulizer does not convert sample to aerosol form (aspiration ceases) below 4 L/min or above 20 L/min. However, the metal high solids nebulizer performance extended to flow rates below 3 L/min. The upper limit for both configurations (Fig. 4 and Fig. 5) of nebulizer based on the Babington principle was the high pressures required to exceed 40 L/min for the 0.61 mm orifice diameter utilized. This was also the reason for not pursuing the Beckman O_2 - H_2 total consumption burner (as a premix nebulizer) higher than 9 L/min. The pressure required at 35 L/min was 120 psi. for the 0.61 mm Babington orifice. Fig. 7 indicates that no sensitivity improvement is realized above 15 L/min for the high solids nebulizer due to the attainment of constant aerosol

density at higher flow rates. Owens - Illinois indicates that this constant aerosol density is maintained well beyond 100 L/min (5); however, the range above 30 L/min is generally not of great interest in spectrochemical applications. The higher flow aerosol range of the high solids nebulizer is, however, useful between 15 and 25 L/min for several slot burners such as the triple slot Boling head and Instrumentation Laboratories CAT No. 24036-03 high solids single slot nitrous oxide - acetylene burner. The extra large dimensions of the slot openings of these two burner heads make them somewhat dangerous (from a flashback standpoint) when operated in the optimum flow range (10 L/min) of the conventional Varian Techtron nebulizer unless auxiliary oxidant is provided. An extra margin of safety is allowed without use of auxiliary oxidant for these specialized burner heads by operating the new high solids nebulizer in the higher flow aerosol region between 15 and 25 L/min where the performance is generally improved as compared to the Varian Techtron nebulizer (Fig. 7). A safer high solids air- acetylene head (1) was, however, used in the present investigations. The nebulizers were therefore operated at 12 L/min for all subsequent studies not concerned with operational range.

At flow rates below 2 L/min, the pressure in the 0.61 mm orifice high solids nebulizers evaluated in these studies is no longer sufficient to rupture the sample film at high enough velocity to generate significant

aerosol. The earlier mentioned smaller orifices will further extend the low flow rate performance, however this was not deemed necessary for the present slot burner studies. A 0.61 mm single hole orifice was therefore used for all subsequent flame studies. The large dynamic operating range of the Babington approach represents an advantage in terms of applicability to a wider variety of spectrochemical situations and reduced susceptibility to gas flow rate errors.

B. Nebulizer Efficiency. Fig. 8 illustrates the effect of the sample uptake rate on the steady state (unlimited sample available) concentration sensitivity of spectrochemical analysis for several nebulizer configurations. Despite the superior operating range, superior tolerance of complex materials, and adequate sensitivity realized with the new high solids approach, Fig. 8 illustrates the principal drawback (low efficiency in terms of sample consumption) of this nebulizer in comparison to the capillary pneumatic system. Although good concentration sensitivity is acheived utilizing the metal adaptation of the Babington principle, Fig. 8 shows that 20-50 mL/min of sample must be delivered to realize that sensitivity at the present state of development of the high solids nebulizer approach. Capillary (premix) nebulizers are normally operated at 5 mL/min. Fig. 8 also illustrates, however, that the non-recirculated 6 mm metal version (Fig. 5) of high solids nebulizer represents a dramatic efficiency improvement over the 16 mm glass respiratory nebulizers including

even the most efficiently recirculated version. The authors attribute this to the lower surface area of the smaller (6 mm) metal hemisphere and to its non-recessed orifice. Further reduction of the metal hemisphere diameter to 3 mm, however, did not further improve the efficiency or operating range. The recessed orifice of the glass configurations (Figs. 1 - 4) may result in thicker sample films in the region of the orifice. This may contribute to formation of more large wasted droplets (that settle out in the premix chamber) during the aerosol generation process. Larger uptake rates then appear to be needed to make up a density of the useful small droplets ($\leq$ 5 µm) similar to that realized with the newly developed, more efficient (non-recessed orifice) metal adaptation of the Babington principle.

C. Hydrodynamic Effects. Fig. 8 also illustrates a hydrodynamic effect of the metal high solids nebulizer. At solution uptake rates below 40 mL/min, a sample delivery gap of 5 mm leads to discrete "dropping" of the sample onto the nebulizer insert. Once a discrete drop is formed, its surface tension appears too great to be overcome as it strikes the top of the pressurized nebulizer hemisphere. Film formation is thereby inhibited and aerosol formation is subsequently depressed. Narrowing the sample delivery gap prevents "dropping" by favoring unbroken "streaming" of the sample onto the nebulizer hemisphere surface. Film formation is more highly favored in this case.

Fig. 9 further demonstrates the "dropping" phenomena tend to be encountered at either excessivly low sample delivery rates or large sample delivery gaps. The most profitable way to avoid this problem is to maintain a sample delivery gap $\leq$ 2 mm which will still allow ready passage of all high solids samples discussed previously (1).

D. Minimum Sample Size Requirements. Fig. 8 is concerned with the spectrochemical response to unlimited sample as a function of uptake rate. Fig. 10, however, illustrates the effect of pipetting limited discrete quantities of solution into the sampling cone of the high solids nebulizer configuration of Fig. 5 as a function of solution uptake rate. Fig. 10 demonstrates that, although 40-50 mL/min is the optimum uptake rate when $\geq$ 2 mL sample are available, slower uptake rates maximize the transient spectrochemical response to smaller samples. In fact a 25-30% sacrifice of concentration sensitivity may be highly desirable with the new high solids approach in order to operate at 20-25 mL/min. Fig. 10 indicates that a 500 µL sample will (at this uptake rate) give a corresponding transient response (0.19 absorbance units) similar to the concentration sensitivity advertised for a Perkin Elmer nebulizer (see Fig. 7).

It should also be noted that the popular steady-state type of absorbance response may also be readily produced using 1 mL of sample and a lesser (12 mL/min) uptake rate to give a 5 sec (reduced sensitivity)

steady-state reading of 0.21 absorbance units (see Fig. 10); 2 mL will give a 10 sec reading of 0.2 A, etc. These reduced steady-state concentration sensitivities, however, still compare favorably with the range (0.18-0.24 A) listed for capillary pneumatic nebulizers by Jarrell Ash and Perkin Elmer Corporation except that about 5 mL/min and 0.4 mL of sample is used with the capillary approach to give a 5 sec steady-state reading. Under these favorable conditions, the required steady-state sample size for the new high solids nebulizer is only 2.5 times more than the required amount for steady-state sampling with capillary pneumatic nebulizers.

Transient sampling, however, represnts a somewhat different situation. In this case, Fig. 11 illustrates that, when all pertinent parameters are optimized, the capillary pneumatic nebulizer approaches its maximum concentration sensitivity with samples as small as 0.1 mL compared to 1 mL required for the metal adaptation of the Babington approach (non-recirculated) and 7 mL for the most efficiently recirculated respiratory glass version. Fig. 12 represents an operational curve used in the laboratory for selection of uptake rates for the metal high solids nebulizer once a sample size is chosen *for transient sampling*. If a 1 mL sample size is chosen, then 40 mL/min will produce an optimum transient of 0.3 A/ppm Cd. The same 1 mL sample may alternatively be used at 12 mL/min (Fig. 10) to produce a 5 sec steady state at $\sim$ 30% less sensitivity.

Table I summarizes the sample size requirements of the spectrochemical adapations of the Babington principle developed in these studies for the production of aerosols from "high solids" materials. The table demonstrates that a 500-fold overall improvement in sample size requirement has been achieved in these studies in comparison with the original respiratory nebulizer approach. The difference in sample size requirements (Table I) for the configurations of Fig. 3 and 4 suggest that recirculation is only significantly beneficial if it is carried out in such a way as to collect both the larger spray droplets (that settle out in the chamber) and the nebulizer sphere runoff. This suggests that, although sphere or hemisphere runoff (back and sides) plays a role, a major mechanism of sample waste is through the large spray droplets produced in addition to small (< 5 μm) aerosol droplets. These larger spray droplets settle out and are either recirculated (Figs. 1 and 4) or exit via the drain (Figs. 2, 3, and 5). In the interest of simplifying the design, reducing memory and sample cross contamination effects, and in consideration of the earlier report (1) of particle size fractionation that may occur in some high solids samples, recirculation has now been abandoned in favor of the approach shown in Figs. 5 and 6. The real improvement of the smaller metal hemisphere with non-recessed orifice is seen in the 30-fold difference in results (Table I) for the configurations of Figures 2 and 5. Although the 1 mL sample size of the high

solids transient or 5 sec steady-state approach at its present stage of development (Figs. 5 and 6) is larger than the 0.1 mL required for transient sampling (0.4 mL for a 5 sec steady state) by the capillary pneumatic nebulizer, a 1 mL sample size requirement is still reasonable for many analytical situations where sample complexity (e.g. Fig. 6) precludes the use of more conventional nebulizers.

E. Impactor Effects. Fig. 13 illustrates the effect of impactor gap on spectrochemical concentration sensitivity. The Varian Techtron capillary nebulizer is seen to require very critical adjustment ($\pm$ 0.2 mm) in comparison to the high solids nebulizer. In fact, the Varian Techtron (adjustable impactor model) capillary pneumatic nebulizer seems to derive its sensitivity advantage (compared to the other commercial nebulizers evaluated) from the precise nature of adjustment provided for the impactor bead gap. Many commercial designs do not include the capability of making such precision adjustments. It may, therefore, not be possible to readily achieve the peak sensitivity shown in Fig. 13 on a routine daily basis for capillary pneumatic nebulizers unless a precision adjustable model is available. In contrast, the high solids nebulizer functions almost as well without any impactor at all as it does when the impactor is adjusted with the greatest of care (see Fig. 13).

III. CONCLUSIONS.

The high solids spectrochemical nebulizer based on the Babington principle represents a system of similar concentration sensitivity, similar precision, superior operating range, simpler design, and far superior sample handling (high solids) capability as compared to the capillary pneumatic approach. High density, finely dispersed aerosols may be produced from solutions and slurries of elevated salt concentration, high solids content, and increased viscosity (1). The parameter to be improved on, however, is the required sample size. Although the present 1 mL transient signal represents a considerable improvement over earlier high solids adaptations of the Babington principle, further improvements are needed to reduce the sample size requirement. At its present state of development, the high solids approach may be optimally used as an auxiliary nebulizer for analysis of those materials ($\geq$ 1 mL) which are too complex to pass through the capillary orifice of a standard pneumatic nebulizer. The high solids approach diagrammed in Fig. 5 and implemented in Fig. 6 is proving to be a simple and convenient "clog-free" approach to spectrochemical sample introduction of highly complex clinical and environmental materials when sufficient sample is available.

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Table I. Sample Size Requirements of Several Spectrochemical Adaptations of the Babington Principle.

Configuration	Figure No.	Minimum Sample Size (mL)
3 mm metal hemisphere	(configured as in 5)	1
6 mm metal hemisphere	5	1
16 mm glass (recirculated) sphere	4	7
16 mm glass (recirculated) sphere	3	22
16 mm glass sphere	2	30
dual 16 mm glass ("HYDROSPHERE") spheres	1	500

Fig. 1. Complete "HYDROSPHERE" Respiratory Nebulizer Configuration (Babington Principle).

a. single post aerosol generator (16 mm glass sphere with recessed orifice -- 0.61 mm hole): b. double post aerosol generator (two 0.43 mm recessed holes); c,d. oxidant gas inlets (rear entry -- not shown); e. modified sample change port.

Fig. 2. Flow-Through Adaptation of "HYDROSPHERE" (Babington Principle Nebulizer for Spectrochemical Analysis.

a. single post aerosol generator mount from "HYDROSPHERE" system; b. 16 mm pressurized glass sphere with recessed orifice (0.61 mm hole); c. 3 mm plastic impaction sphere, fixed position; d. cylindrical Plexiglas housing for nebulizer inserts based on the Babington principle.

Fig. 3. Recirculated Flow-Through Adaptation of "HYDROSPHERE" (Babington Principle) Nebulizer.

Simplified cross-sectional view: a. single post aerosol generator from "HYDROSPHERE" system; b. conical reservoir (nylon) for sample entry and nebulizer runoff recirculation; c. sample entry (fill) channel; d. nebulizer delivery channel (from reservoir); e. two-way valve; f. sample exit (empty) after reading obtained.

Fig. 4. Increased Recirculation Efficiency Flow-Through Adaptation of "HYDROSPHERE" (Babington Principle) Nebulizer.

Simplified cross-sectional view: a. single post aerosol generator from "HYDROSPHERE" system; b. "half cone" (nylon) for runoff collection (drains into reservoir, c); c. conical reservoir (Plexiglas; sealed to container side) for sample entry and runoff collection from chamber walls (condensed large droplets) as well as nebulizer sphere; d. conical Plexiglas ceiling to minimize large droplet hangup. Conical shape allows condensed droplets to flow more readily down to recirculation reservoir (c); e. sample entry (fill) channel; f. nebulizer delivery channel (from reservoir); g. two-way valve; h. sample exit (empty) after reading obtained.

Fig. 5. Improved Flow-Through Adaptation of the Babington Principle for Spectrochemical Analysis (No Recirculation).

a. interchangeable 6 mm o.d. or 3 mm o.d. pressurized closed-end polished metal tube nebulizer insert with non-recessed orifice (0.61 mm hole); b. 3 mm plastic impaction sphere (translational movement possible); c. mounting block with internal rotary-translational motion converter for impactor movement; d. sample delivery tube (2.38 mm i.d.); e. modified compression "elbow" fitting; f. external adjustment for impaction gap; g. set screw to hold compression elbow in place; h. "pop off" safety plug (in case of burner flashback).

Fig. 6. Finely Dispersed Tomato Sauce Aerosol Emanating from 10 cm Slot Burner.

Direct aerosol production (using system diagrammed in Fig. 5) is seen to be easily accomplished without system clogging. Burner is shown without flame burning to demonstrate high density finely dispersed aerosol.

Fig. 7. Nebulizer Operating Range.

○ Varian Techtron capillary nebulizer; □ High solids nebulizer, 6 mm metal; ◺ High solids nebulizer, 16 mm glass respiratory; △ Beckman total consumption burner as premix nebulizer.

Fig. 8. Nebulizer Efficiency Comparison.

○ Varian Techtron capillary nebulizer; △ High solids nebulizer, 6 mm metal, non-recirculated, 5 mm gap; □ High solids nebulizer, 6 mm metal, non recirculated, 0.6 mm gap; ◺ High solids nebulizer, 16 mm glass respiratory, recirculation of all excess.

Fig. 9. Sample Delivery Gap Effects.

Sample Delivery Rate: △ 53 mL/min; ○ 21 mL/min; □ 10 mL/min; ◺ 4.5 mL/min.

Fig. 10. Sample Size and Uptake Rate Requirements.

Sample Size: -- unlimited; ○ 2 mL; ◺ 1 mL; △ 0.5 mL; □ 0.2 mL; ◺ 0.1 mL.

Fig. 11. Comparison of Minimum Required Sample Volume.

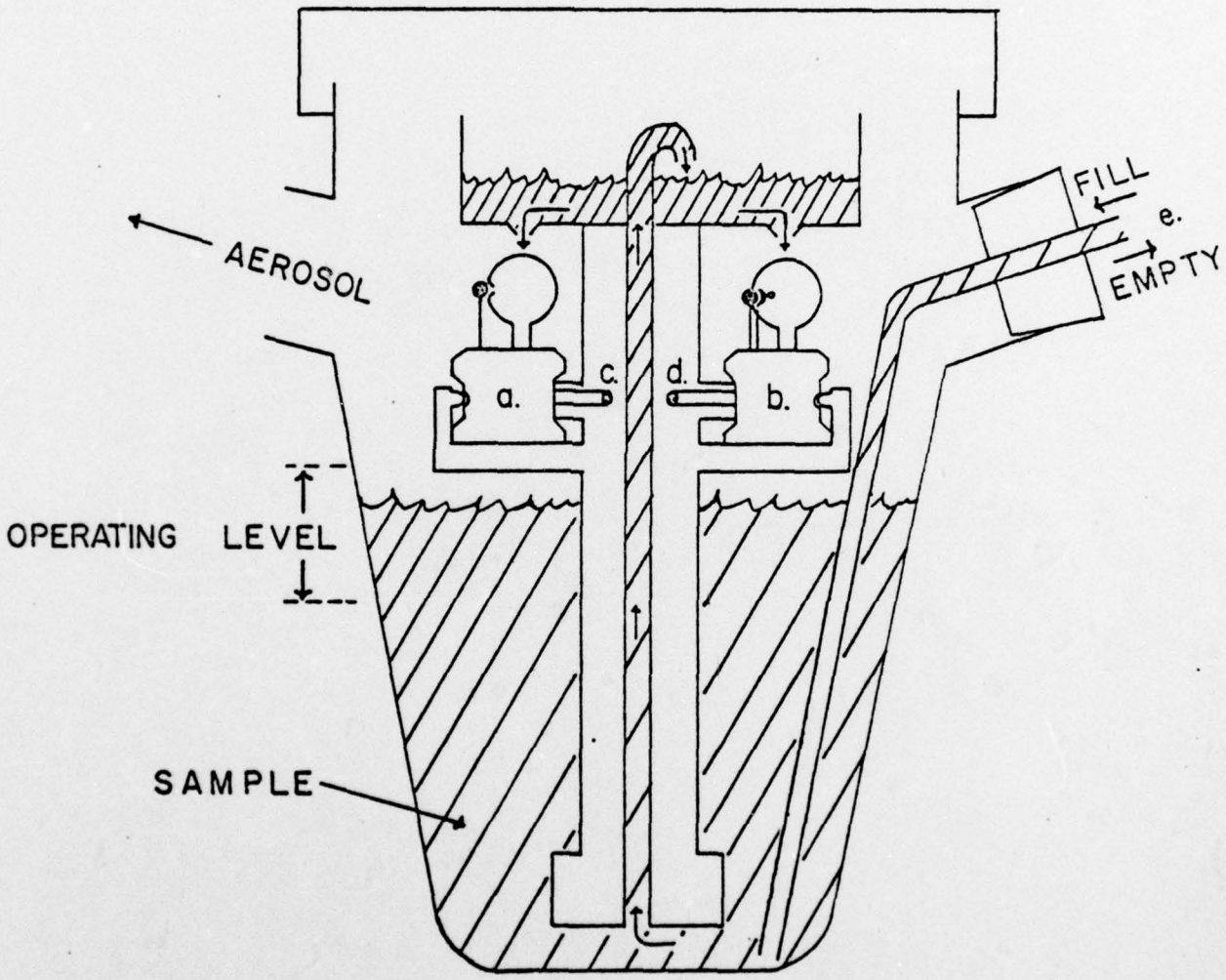
- ○ Varian Techtron capillary nebulizer;
- △ Jarrell Ash (Hetco) capillary nebulizer;
- □ High solids nebulizer, 6 mm metal, non-recirculated;
- ▯ High solids nebulizer, 16 mm glass respiratory, recirculation of all excess.

Fig. 12. Operational Curve of Sample Volume-Uptake Rate (Optimum).

Note that the ordinate represents flow rate. This figure does not reflect on the sensitivity at a given volume.

Fig. 13. Impactor Effects.

- ○ Varian Techtron capillary nebulizer;
- □ High solids nebulizer.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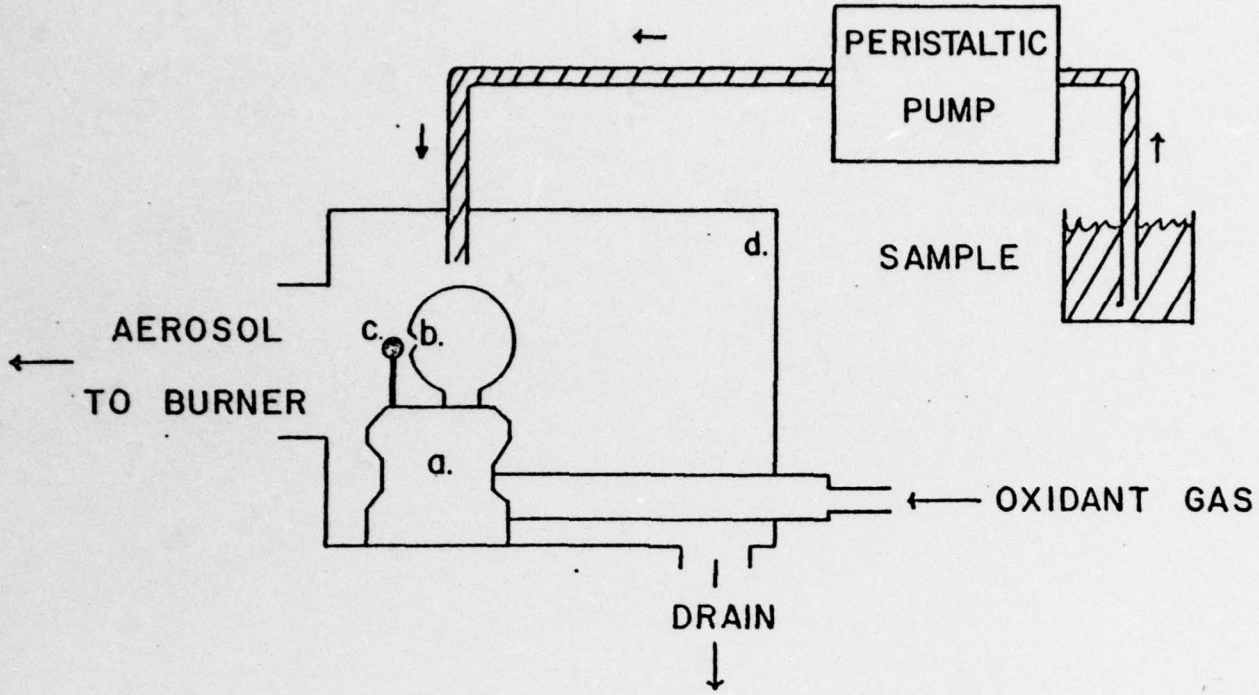
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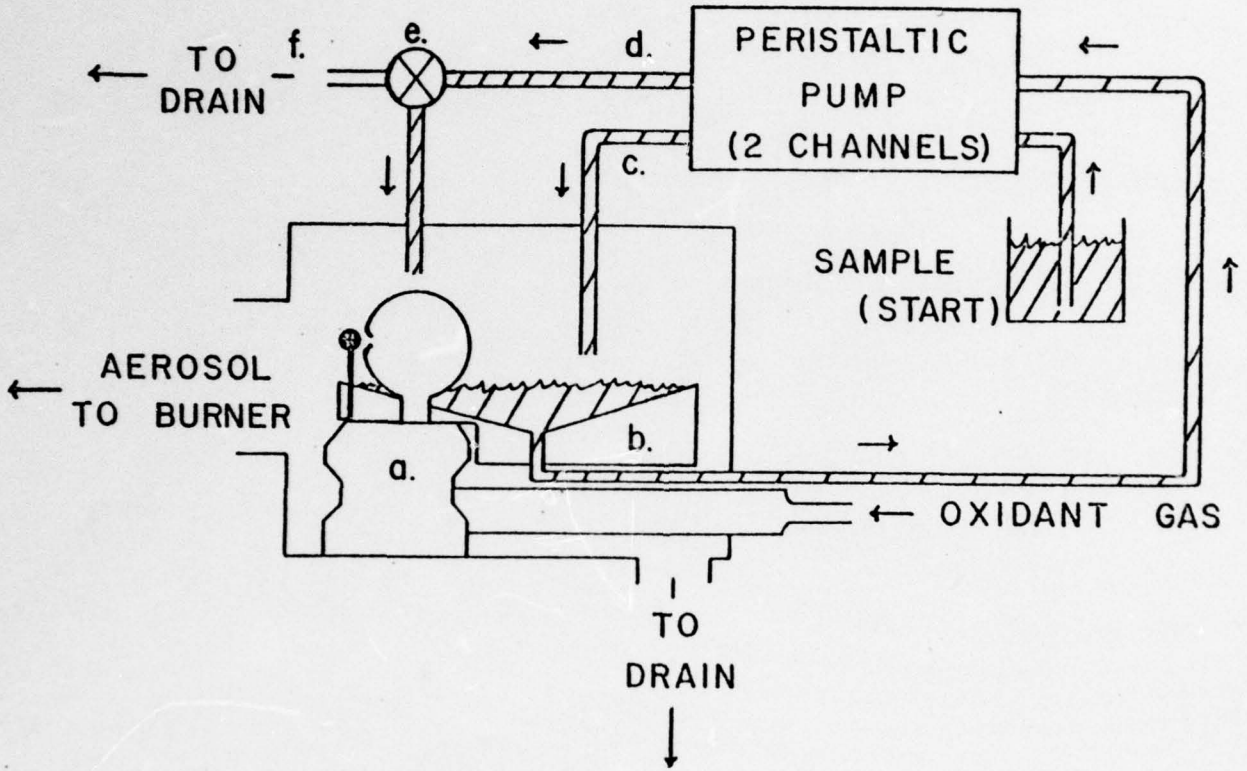
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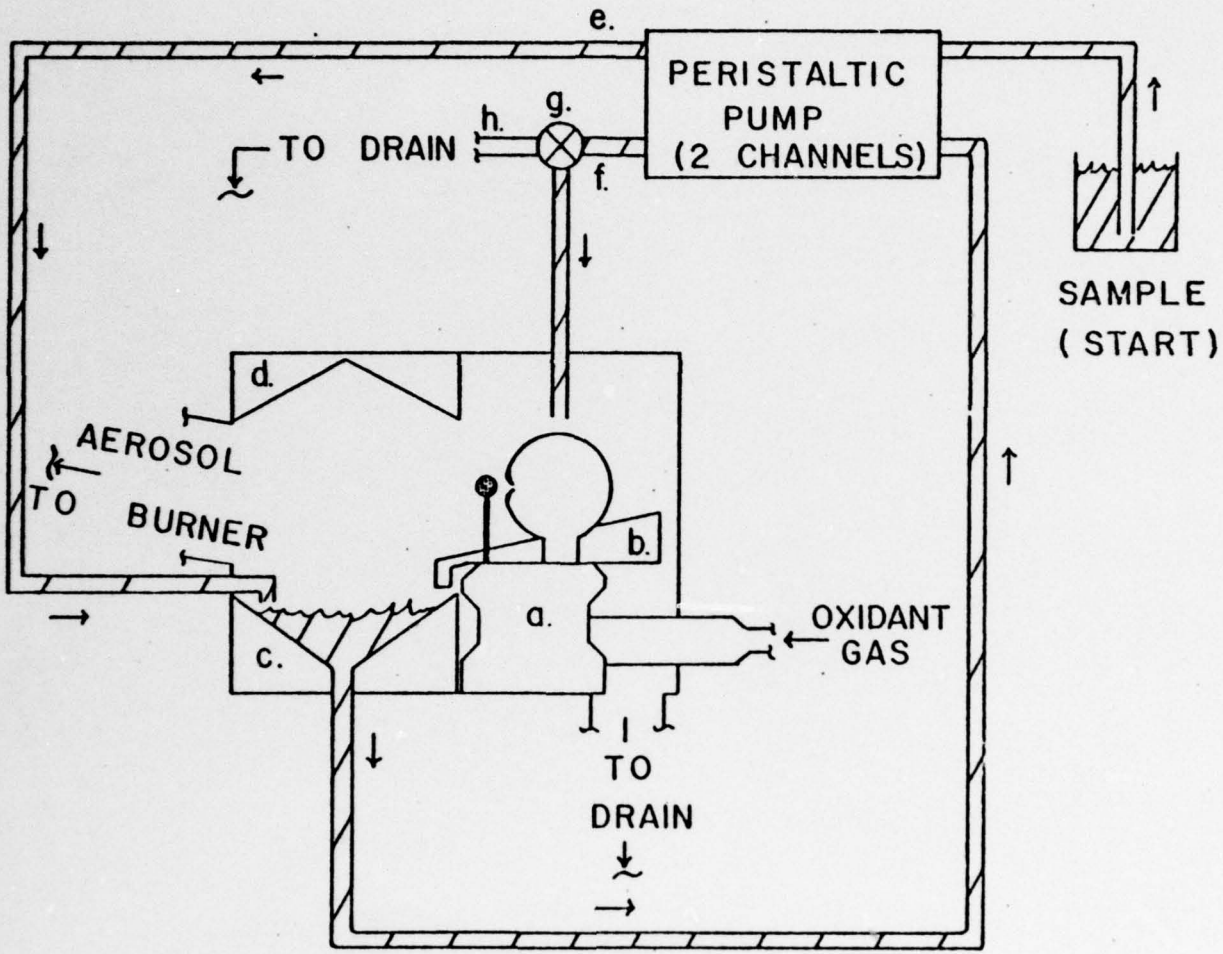
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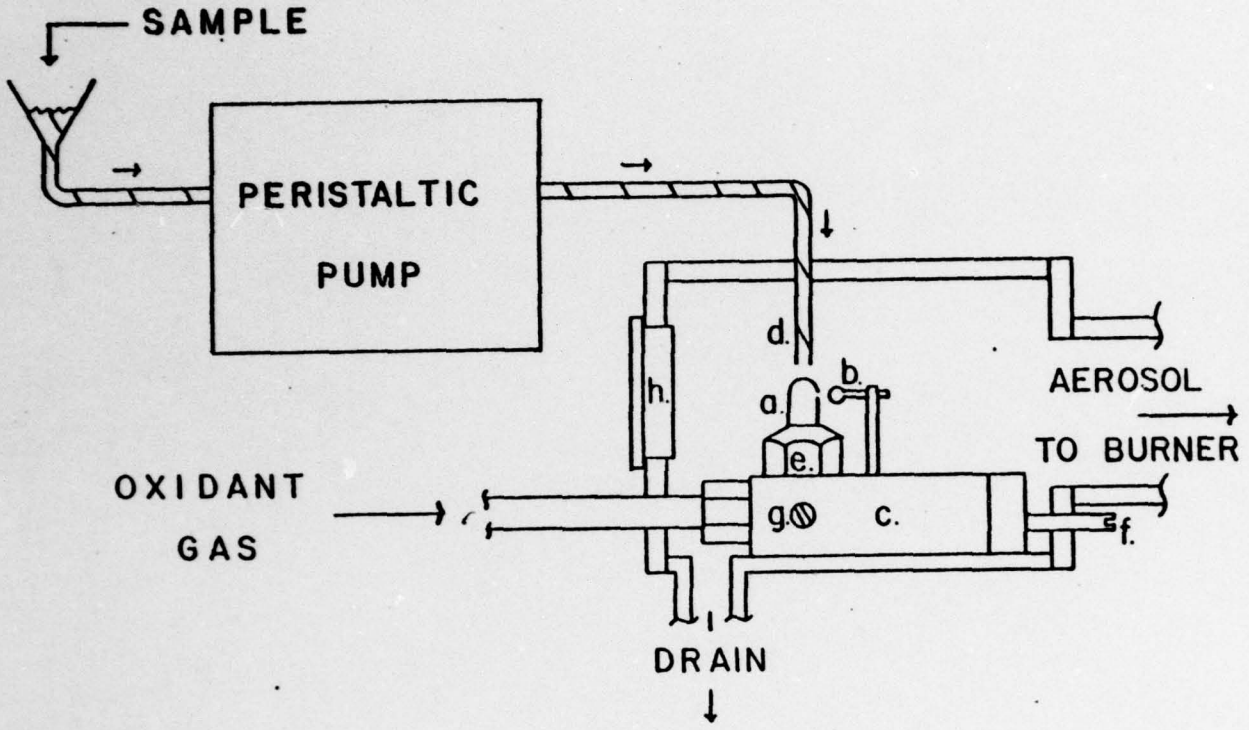
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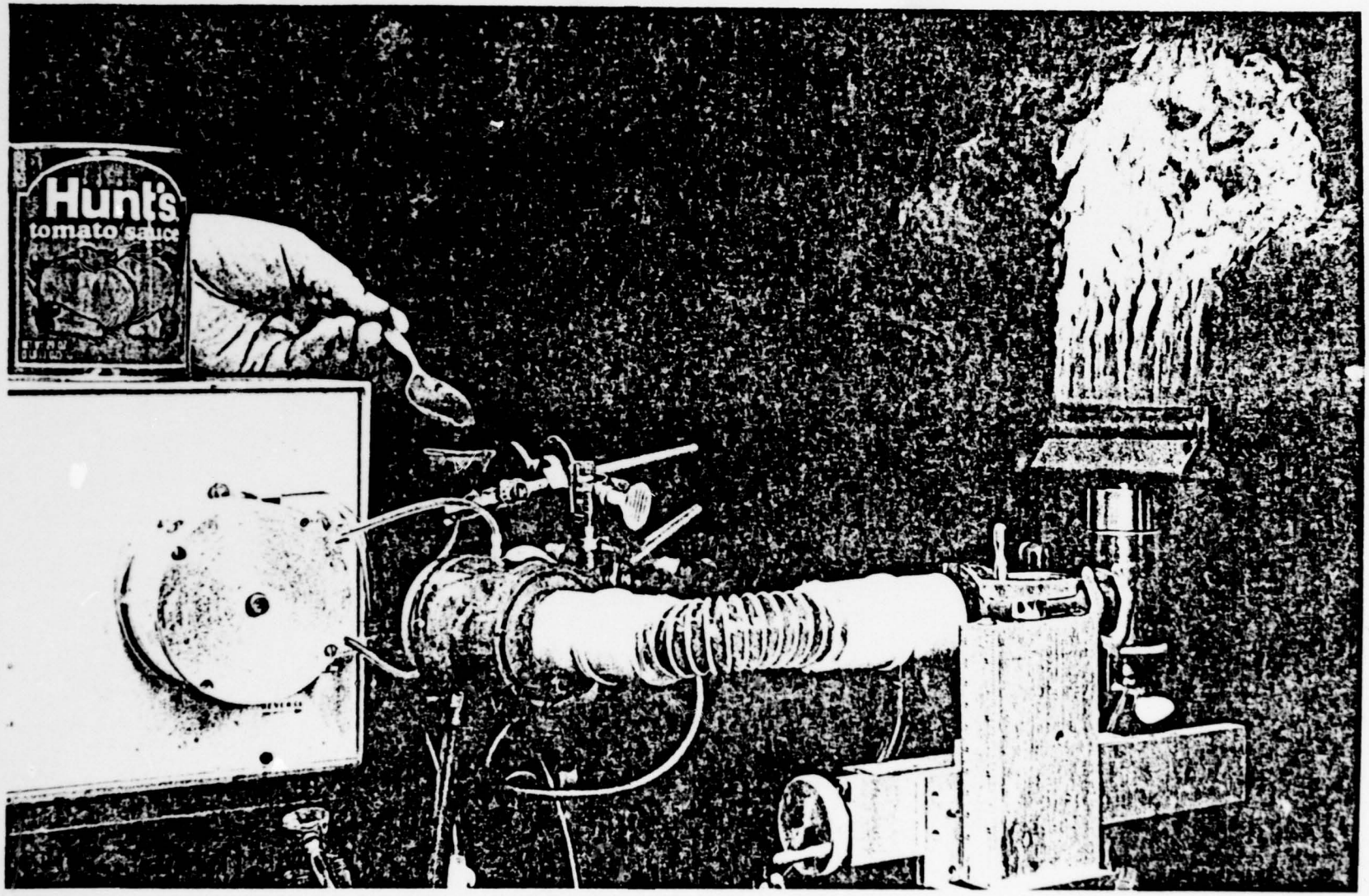
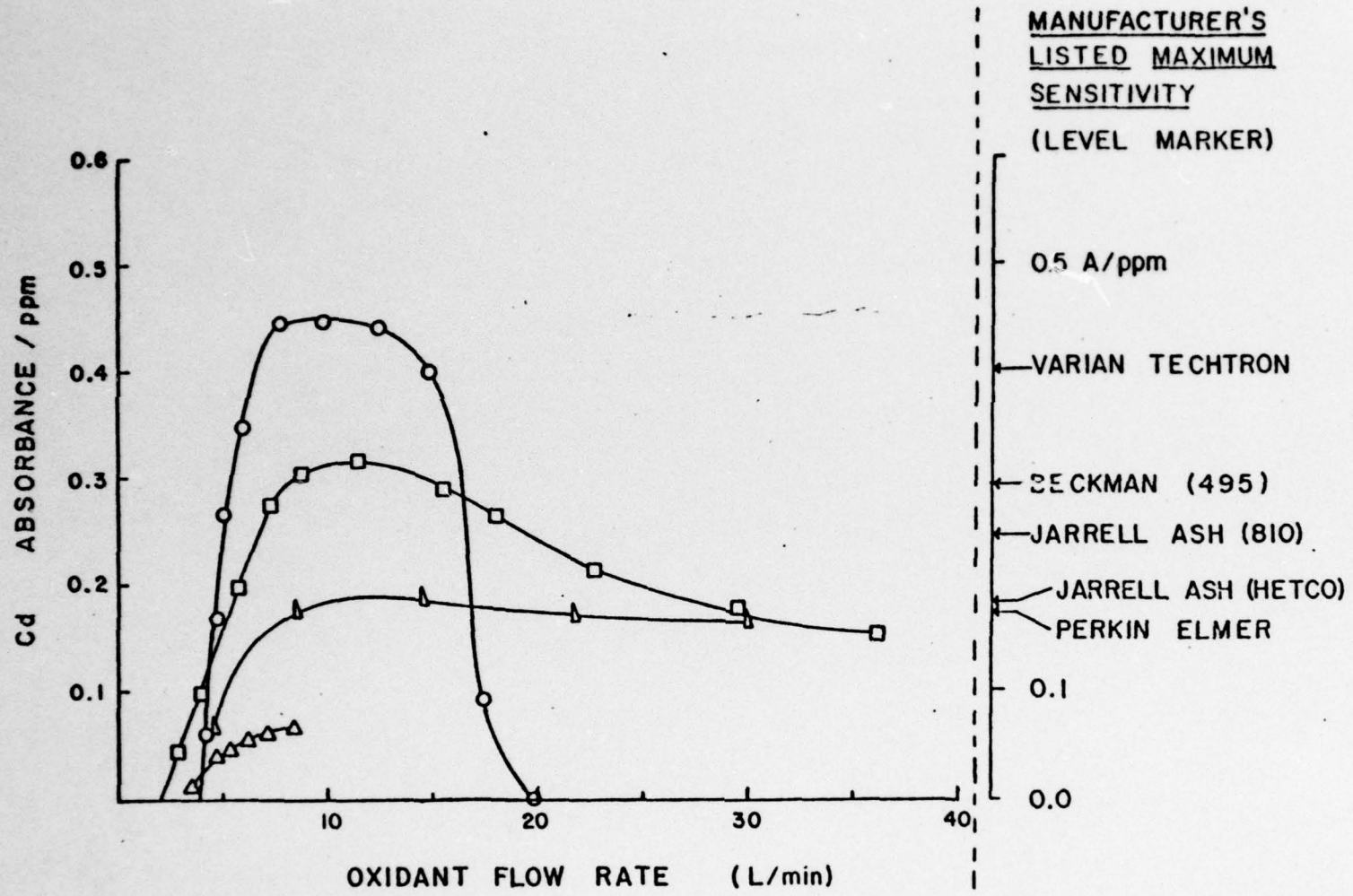
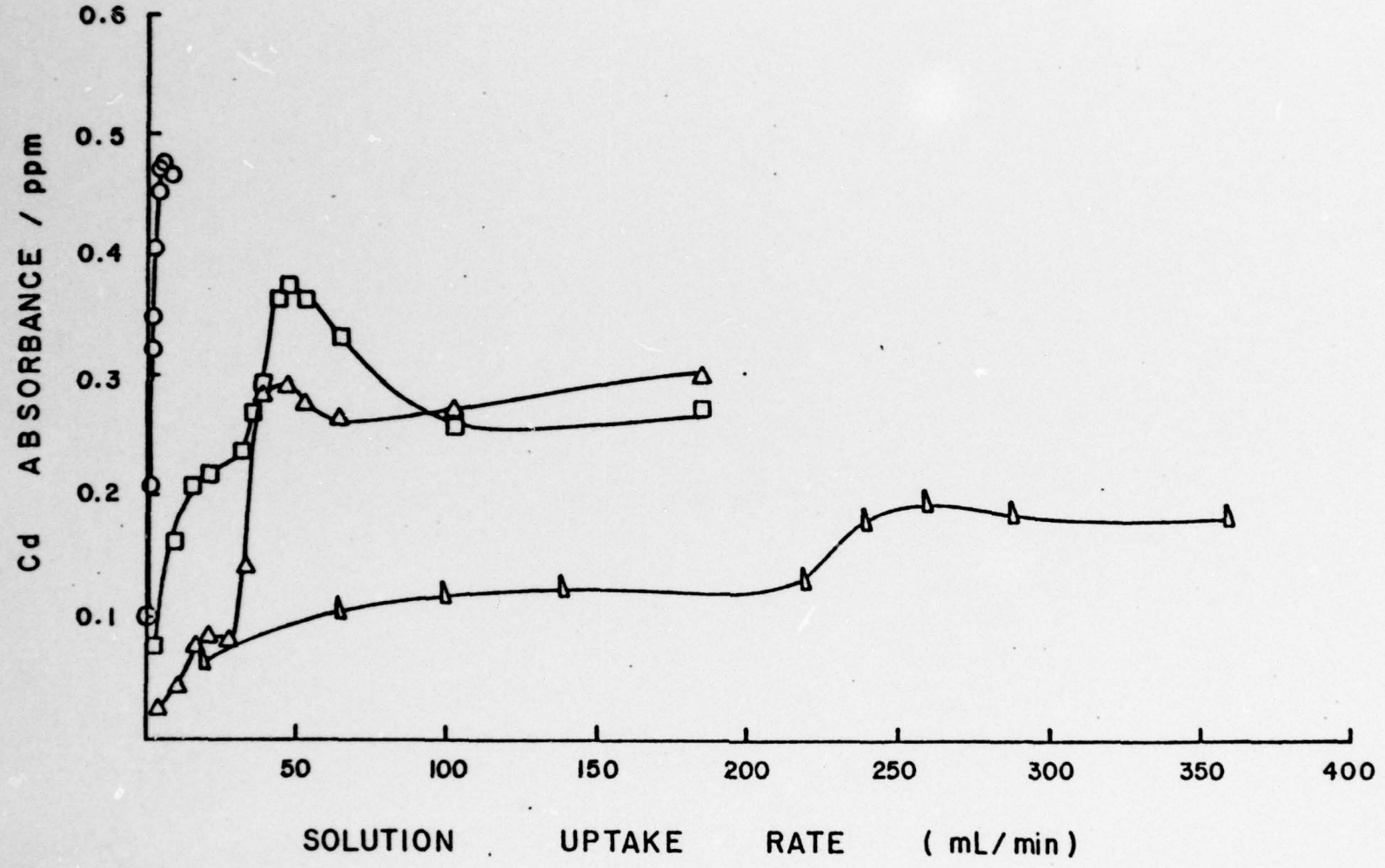
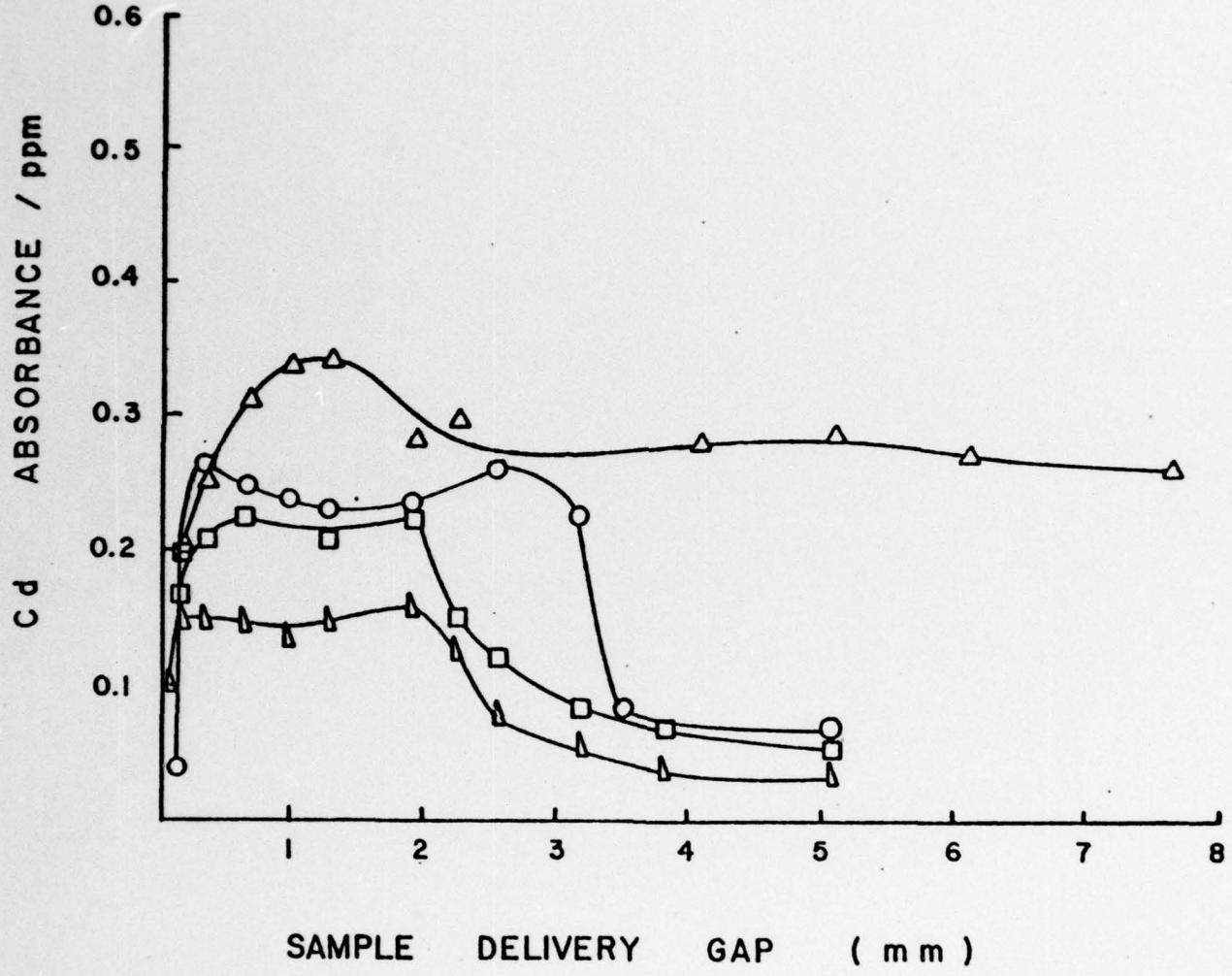
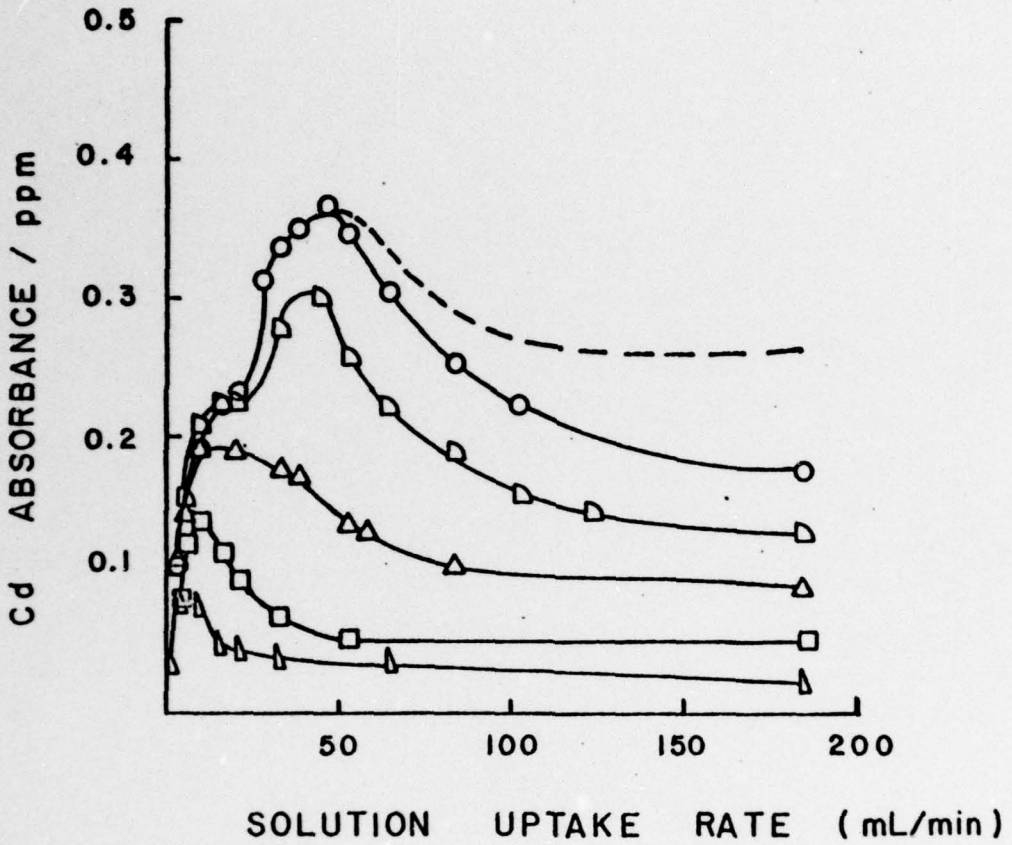
DRAIN

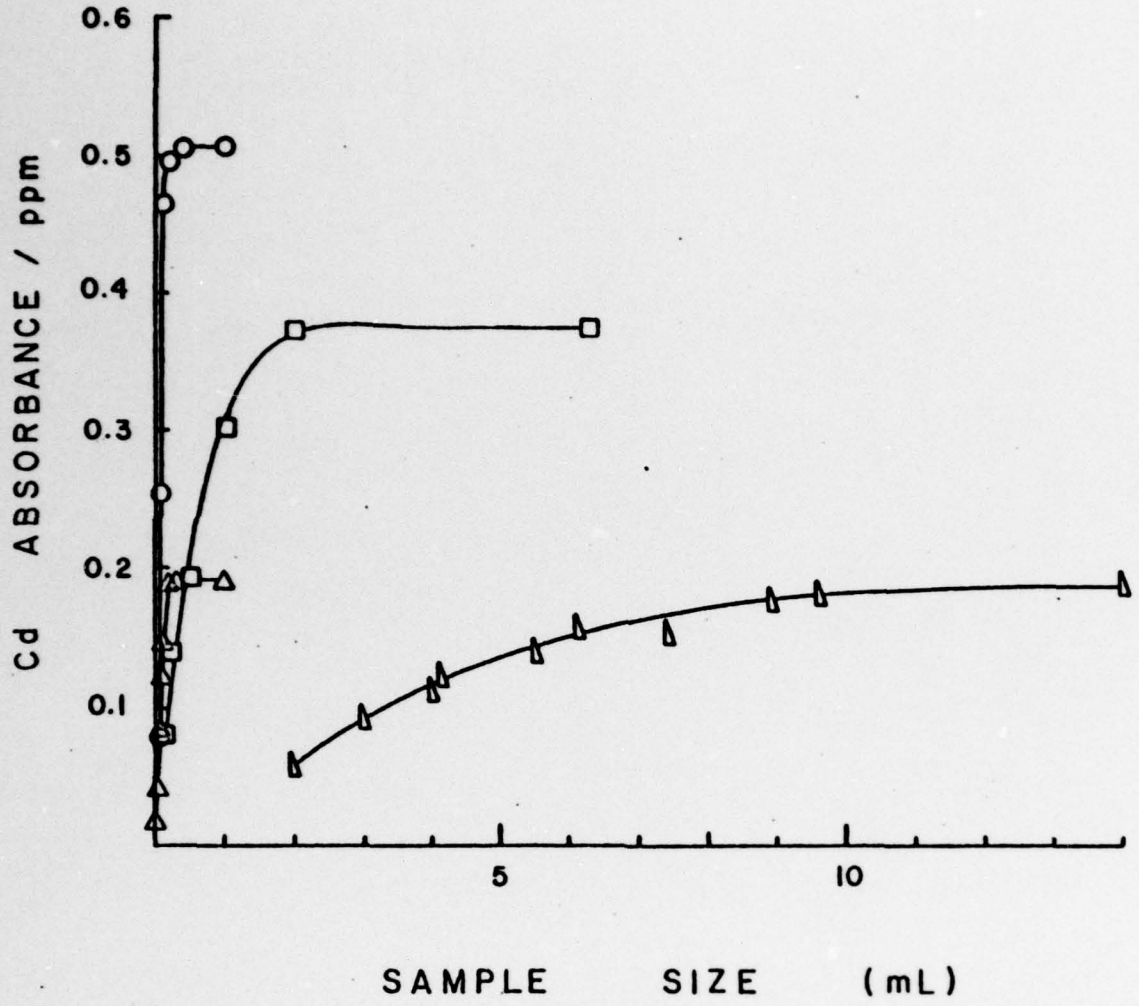

Hunt's
tomato sauce

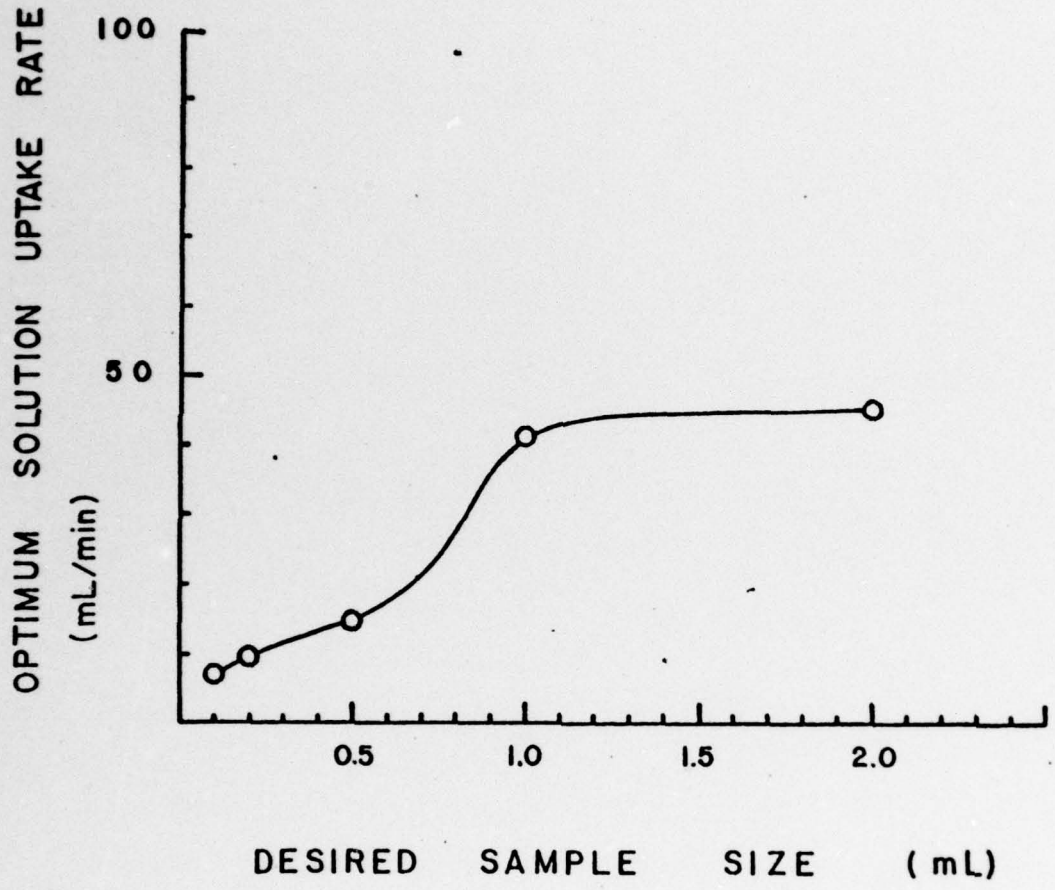

MANUFACTURER'S
LISTED MAXIMUM
SENSITIVITY
(LEVEL MARKER)
0.5 A/ppm
VARIAN TECHTRON
BECKMAN (495)
JARRELL ASH (810)
JARRELL ASH (HETCO)
PERKIN ELMER
0.1
0.0
Cd ABSORBANCE / ppm
0.6
0.5
0.4
0.3
0.2
0.1
10
20
30
40
OXIDANT FLOW RATE (L/min)

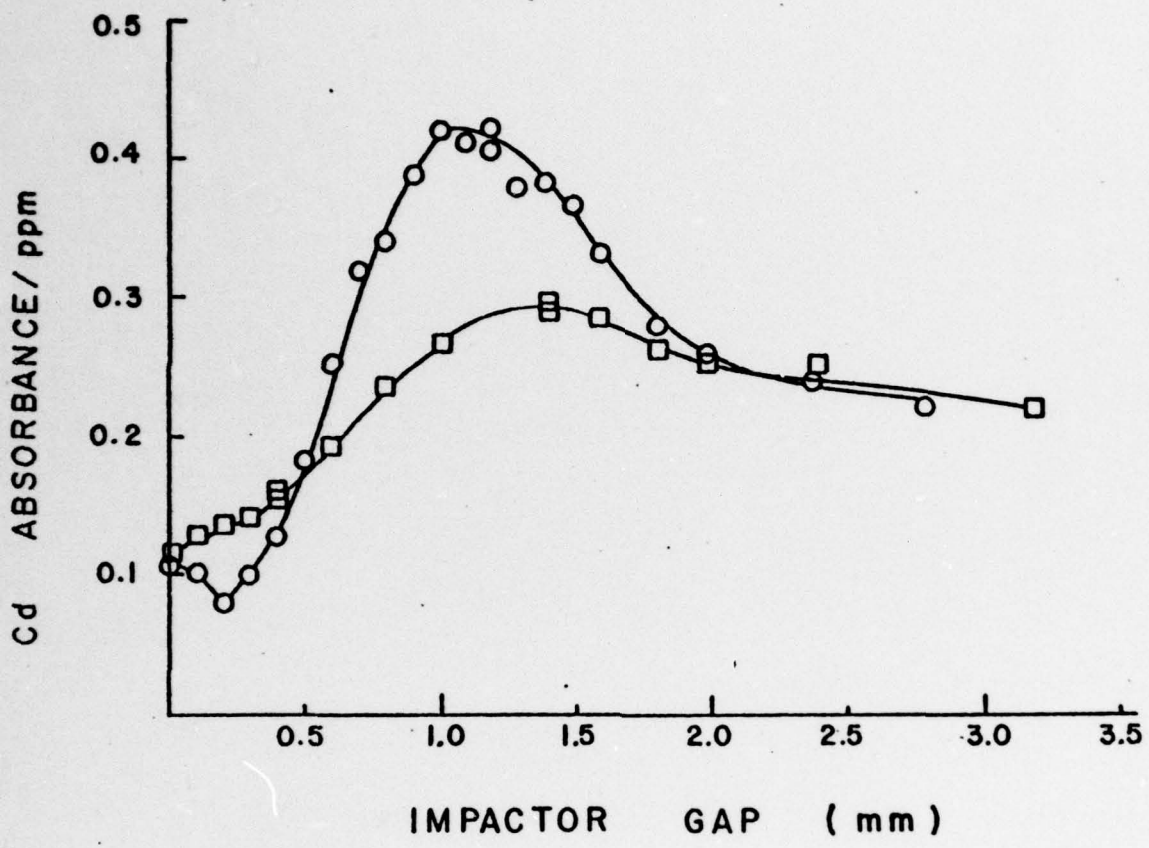

0.6
0.5
0.4
0.3
0.2
0.1
Cd ABSORBANCE / ppm
50
100
150
200
250
300
350
400
SOLUTION UPTAKE RATE (mL/min)


0.6
0.5
0.4
0.3
0.2
0.1
Cd ABSORBANCE / ppm
1
2
3
4
5
6
7
8
SAMPLE DELIVERY GAP (mm)


0.5
0.4
0.3
0.2
0.1
Cd ABSORBANCE / ppm
50
100
150
200
SOLUTION UPTAKE RATE (mL/min)


0.6
0.5
0.4
0.3
0.2
0.1
Cd ABSORBANCE / ppm
5
10
SAMPLE SIZE (mL)


OPTIMUM SOLUTION UPTAKE RATE
(mL/min)
100
50
0.5
1.0
1.5
2.0
DESIRED SAMPLE SIZE (mL)


0.5
0.4
0.3
0.2
0.1
Cd ABSORBANCE/ ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
IMPACTOR GAP (mm)

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